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INSTRUCTIONS FOR THE USE OF A SEDIGRAPH 5000ET PARTICLE SIZE ANALYSER, WITH AN APPLE IIe COMPUTER

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April 1987

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Project: 30.13.03
Evaluation of Mineral Processing Equipment

22 pp

MINERAL SCIENCES LABORATORIES
DIVISION REPORT MSL 87-61 (TR)

MICROMEDIA

INSTRUCTIONS FOR THE USE OF A SEDIGRAPH 5000ET PARTICLE SIZE ANALYSER, WITH AN APPLE IIe COMPUTER

by

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ABSTRACT

A set of instructions have been prepared for the use of the Sedigraph 5000ET particle-size analyser, with an Apple IIe computer. These instructions are simplified for those unfamiliar with computer-aided techniques.

The Sedigraph can accurately measure particle sizes down to 0.1 μm, the range commonly termed 'sub-micron' or 'slimes'. With the assistance of the Apple IIe computer, data can be presented in various forms. Examples of print-out data for deslimed talc and kaolin are presented in the Appendix.

The Sedigraph compares favourably with other fine-particle size analysers.

Keywords: Instructions, sedigraph, particle-size analysis, fine particles, computer-aided.

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MODE D'EMPLOI DE L'ANALYSEUR GRANULOMÉTRIQUE SÉDIGRAPHE 5000ET AU MOYEN D'UN ORDINATEUR APPLE IIe

par

P.R.A. Andrews*, J.M. Lamothe** et R.D. Joe***

RÉSUMÉ

Une série d'instructions ont été rédigées pour l'utilisation de l'analyseur granulométrique Sédigraphe 5000ET au moyen d'un ordinateur Apple IIe. Ces instructions sont simplifiées pour ceux qui ne connaissent pas bien les techniques assistées par ordinateur.

Le Sédigraphe peut mesurer avec précision la taille des particules allant jusqu'à 0,1 µm, l'échelle communément appelée "submicron" ou "schlamms". À l'aide de l'ordinateur Apple IIe, les données peuvent être présentées sous différentes formes. Des exemples de sorties de données pour le talc et le kaolin déschlammés sont présentés en annexe.

Le Sédigraphe se compare favorablement aux autres analyseurs granulométriques de particules fines.

MOTS-CLÉS: Instructions, sédigraphe, analyse granulométrique, particules fines, assistance par ordinateur.

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INTRODUCTION

A Micromeritics model 5000ET automatic sedimentation particle size analysis system was purchased in September 1984. An Apple IIe computer, with ancilliary software, also was purchased to facilitate the automatic calculation of size analysis.

The Sedigraph 5000ET was developed to quickly and accurately measure incremental particle size of a fine multi-sized sample dispersed in a sedimentation liquid. The size analysis is plotted automatically as cumulative weight percent versus particle diameter. The instrument is easily programmed for various mineral* specific gravity values, sedimentation liquid densities and viscosities. The Sedigraph can operate unattended and resets automatically.

PRINCIPLE OF OPERATION

The Sedigraph particle size analyser measures the sedimentation rates of particles dispersed in a liquid, according to Stokes Law:

$$V_t = \frac{g\, d^2(\sigma - p)}{18\eta}$$

where V_t = terminal velocity
g = acceleration due to gravity
d = particle diameter
σ = particle specific gravity
p = liquid specific gravity
η = liquid viscosity

The data are automatically plotted as cumulative weight percent versus equivalent spherical diameter, i.e., the diameter of a sphere of the same material which falls under gravity with the same velocity as the particle. The instrument uses a finely collimated beam of low-energy X-rays to measure

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* Both metallic and non-metallic minerals are implied.

the concentration of particles. A solid-state digital sequencer controls all instrument functions to continuously solve Stokes Law relationships.

The Sedigraph can measure the size of most mineral powders with a particle size range of 100 to 0.1 μm. The mineral to be sized must also satisfy three general criteria; the mineral specific gravity must be > 1.0; the mineral must be more absorptive of X-rays than the dispersion liquid; and the mineral particles must be completely dispersed. The first two criteria should not cause any difficulty; the dispersion properties of minerals do, however, vary.

Generally, water or a 5% solution of sodium silicate, with the aid of mechanical stirring in a beaker, should be sufficient to disperse the mineral particles. With some minerals it may be necessary to use other dispersing agents; on occasion a surfactant such as Aerosol OT and a mechanical blender or ultrasonic bath aid dispersion. Use of a surfactant is necessary when sizing flotation products which have previously been chemically treated with organic collectors. A summary of some dispersing fluids for non-metallic minerals is presented in Table 1.

OPERATING PROCEDURE

Instructions for use of the Sedigraph and the Apple IIe computer are provided in the manufacturers operating manual. The computer-Sedigraph system was designed to facilitate the calculation of parameters and/or the manipulation of information produced by the Sedigraph. The operator should be aware, however, that the computer does not control or set the Sedigraph. The following is a simplified description to assist those who may be unfamiliar with computer operation.

Preliminary

On the Sedigraph:

1. X-Ray and FLOW switches OFF.
2. MASTER switch ON, the green light should glow.
3. Verify compatibility on the tubing with proposed liquid.

4. Add liquid in beaker if necessary.
5. Activate pump by switching FLOW ON.
6. Eliminate trapped air bubbles and possible sediments.
7. Close door and turn X-Ray key-type switch ON.
8. Verify X-Ray intensity and adjust if necessary by using the INTENSITY dial. The intensity should be between 40 and 50 on the meter. A Low/High intensity switch is also situated inside the compartment.
9. Wait 30 min. before use.

On the Computer:

10. Ensure that the CAPS LOCK key is on upper letter position.
11. Insert diskette labelled PROGRAM MASTER in disk drive No. 1.
12. Turn ON main switch on the left side of the computer.
13. When] appears on the lower left of the screen, press CONTROL and RESET keys simultaneously.
14. Type RUN and press RETURN key.
15. The MAIN MENU will then appear:

    1. OPERATING PARAMETERS
    2. INITIATE ANALYSIS
    3. REPORT PROGRAMMING
    4. DATA FILES

Remove the PROGRAM MASTER diskette and replace with DATA MASTER diskette in drive No. 1. A specific mode may now be invoked by pressing the corresponding number.

OPTIONS Description

1. OPERATING PARAMETERS

Once this option is invoked, the operator has two alternatives:

1.1 Press E to invoke edit mode i.e. to set necessary parameters

1.2 Press M to go back to MAIN MENU

The following required information should then be completed when in EDIT mode.

DATE:

OPERATOR:
SAMPLE:
SAMPLE DENSITY:
LIQUID:
TEMPERATURE:
LIQUID DENSITY:
LIQUID VISCOSITY:
PRINCIPAL PARTICLE DIAMETER: (MICRONS)
STARTING: (MICRONS) ENDING: (MICRONS) MAX: (MICRONS)

RUNNING TIME: RATE:

Temperature is located inside the compartment of the Sedigraph. The liquid density and viscosity are available in APPENDIX 4 in the black reference binder or on bottle labels for Sedisperse liquids. STARTING DIAMETER and RATE are automatically calculated and when the C appears in their place, press C for each one.

1.3 The OPERATING PARAMETERS is now out of EDIT mode. To correct any errors or to recalculate using any other set of conditions, the operator should invoke back to EDIT mode and repeat the procedure. Pressing the RETURN key alone will accept the information already written on the line.

1.4 When all information is verified, the operator must return to MAIN MENU by pressing M in order to invoke the next step (or OPTION).

2. INITIATE ANALYSIS

2.1 Check that all values displayed on the screen correspond with the desired values. Check that the RATE dial is set on the Sedigraph.

2.2 Stop the pump on the Sedigraph by switching FLOW OFF.

2.3 Fill the sample beaker with ≈ 25 to 35 mL of clean dispersion liquid and put the magnetic stirrer into the solution.

2.4 Set the FLOW dial on the Sedigraph to the 'ON' position.

2.5 Make sure that there are no entrapped air bubbles in the cell.

2.6 Turn the X-Ray key to the ON position.

2.7 The intensity meter (top right-hand corner) should reach between 40 and 50 DC microamperes when the dispersion liquid solely is present and the X-RAY on. If this does not occur then the INTENSITY dial should be unlocked and reset so that the meter reads between 40 and 50 DC microamperes.

2.8 Ensure that the cell is in the starting position by resetting the RUN switch. This can be done by pushing down and back up to middle position.

2.9 Turn 100 PERCENT dial clockwise as far as possible.

2.10 Adjust 0 PERCENT dial so that the line displayed on the screen: CUMULATIVE MASS PERCENT is 0.

2.11 Add ≈1 g of sample to the beaker and ensure that the magnetic stirrer is rotating.

2.12 Close the door on the Sedigraph and watch CUMULATIVE MASS PERCENT value (displayed on the CRT) decrease to 100, but not below 100.

2.13 Repeat steps 2.11 and 2.12 until 100 or more is reached, but not more than 160.

2.14 Adjust the 100 PERCENT dial so that the value is exactly 100. N.B. The 100 PERCENT dial is invalid when turned more than five times counter clockwise. Do not press F or Z as this is to use both the Sedigraph and the Apple display.

2.15 Press the < SPACE > bar to commence a run.

2.16 Push RUN button up on the Sedigraph to commence the test.

2.17 Let the test run for the desired time. If an error has been made at this point then press A on the keyboard to abort the test and repeat steps where the mistake occurred and start the test again.

2.18 When the test is complete, press T on the keyboard to terminate and push the RUN button on the Sedigraph down to reset and then back up to the middle position. The next mode will be automatically invoked.

3. REPORT PROGRAMMING

3.1 Once this mode is invoked there are ten choices that the operator can choose from but only four can be used at one time.

3.2 Press E to edit the options in this mode

3.3 Press the desired number according to the desired option
Note that the information from a previous run is not erased automatically. The operator should option first CLEAN VIDEO to prevent the previous information from appearing with the new information.

3.3.1 SAVE ANALYSIS DATA - stores information from the test for future reference

3.3.2 SAVE VIDEO DATA - stores what is displayed on CRT for future reference

3.3.3 LOAD ANALYSIS DATA - retrieves stored information from previous tests

3.3.4 LOAD VIDEO DATA - retrieves stored information from previous screens

3.3.5 EXCHANGE BUFFERS - places what is on the screen into memory

3.3.6 PLOT DATA ON CRT - places information in tabular form on the CRT (television)

3.3.7 COPY VIDEO ON LP - prints hardcopy of video display
- will print only if the previous execution line is PLOT DATA ON CRT

3.3.8 PRINT TABLE ON LP - prints hardcopy of tabular results

3.3.9 CLEAR VIDEO - Erases information from a previous run but will not erase information on the diskette

3.3.10 END - completed.

3.4 After necessary options have been made, press X to execute program.

TYPICAL PROBLEMS ENCOUNTERED

1. If a previous operator has used a program which required lower case letters, ERROR SYNTAX will appear when RUN is typed and RETURN is pressed. When this happens press CAPS LOCK and repeat.
N.B. This program uses CAPITAL LETTERS.

2. If the 0 on the computer is difficult to regulate, check that the X-Ray is on. If yes, verify that the magnetic stirrer does not create any vortex and eliminate any air bubbles trapped in the cell.

3. When on INITIATE ANALYSIS option, if no number appears on cumulative percent weight, press CONTROL, OPEN APPLE and RESET keys simultaneously. This will reload the program in the correct way.

4. If the 100 on the computer is not stable, check whether there is a vortex in the beaker (i.e. too violent an agitation), or any sedimentation in the beaker.

5. If any unusual variation happens during a test (a dramatic increase of cumulative percent weight), reset both computer and Sedigraph and check whether there are air bubbles in the cell.

6. If the pump does not start, check that the tubes are not blocked and that the pump case is not too tight.

7. If particles adhere to the inside surfaces of the sample cell, use alcohol to remove them.

PARTICLE SIZE ANALYSIS

When processing minerals it is often necessary to deslime samples by hydrocycloning, a wet classification technique. The success of subsequent beneficiation will depend on the efficiency of the desliming operation. An instrument that can quickly and accurately measure the size of the underflow (coarse product) and overflow (fine product), from a desliming operation is therefore of considerable importance.

The size analyses of the overflow and underflow of ground talc and washed kaolin, as measured by the Sedigraph, are presented in printout form in the Appendix.

CONCLUSIONS

The Sedigraph 5000ET particle-size analyser can accurately measure particle sizes down to 0.1 μm. This is the size range commonly referred to as 'slimes', so that when the Sedigraph is used in conjunction with accurate desliming processes, slime losses can be better controlled by knowing the size of slimes.

The Sedigraph compares favourably with other sub-micron particle-size analysers provided that the sample processed is composed of mineral particles of the same density (or approximately the same density).

ACKNOWLEDGEMENTS

The authors would like to thank L. Morin of the Non-Metallic Minerals Section, Mineral Processing Laboratory, CANMET for particle-size determinations.

Table 1. Dispersing liquids for some non-metallic minerals

Mineral	
Barytes	- 0.1% Calgon in water
Beryl*	- 0.1% sodium silicate in water
	- 0.1% Calgon in water
Carbon Black	- 0.2% tannic acid in water
Carborundum	- 0.2% Calgon in water
Coal	- Ethyl alcohol containing 0.1 mol/L calcium
	- chloride
Dolomite	- 0.1% Calgon in water
Feldspar	- Distilled water
Fluorspar*	- 0.001 M potassium chloride in methyl alcohol
	- 0.1% Calgon in water
Glass powder*	- Butyl alcohol
	- Distilled water
Graphite*	- Ethyl alcohol
	- 0.5 g/L tannic acid in water
	- 0.5% sodium linoteate in water
	- 0.5% (by volume) Aersol OT in water
Gypsum	- 0.1 mol/L calcium chloride in alcohol
Kaolin	- 0.005 mol/L sodium pyrophosphate in water
	- 0.1% Sodium silicate in water
Limestone	- 0.2% sodium silicate in water
	- 0.005 mol/L sodium pyrophosphate in water
Phosphate ore	- 0.1% Calgon in water
Quartz	- Distilled water
Silica	- 0.005 mol/L sodium pyrophosphate in water
	- 1:1 ethyl alcohol to water mixture
Silicate minerals	- 0.005 mol/L sodium pyrophosphate in water
	- 1:1 ethyl alcohol to water mixture
Talc	- 0.1% Calgon in water
Titanium dioxide	- 0.1% Calgon in water

* For some minerals that are different to disperse, several alternate dispersants are suggested.

APPENDIX

OCTOBER 7/1986

OPERATOR: L.MORIN

SAMPLE: TALC **OVERFLOW**
SAMPLE DENSITY: 2.92 G/CC

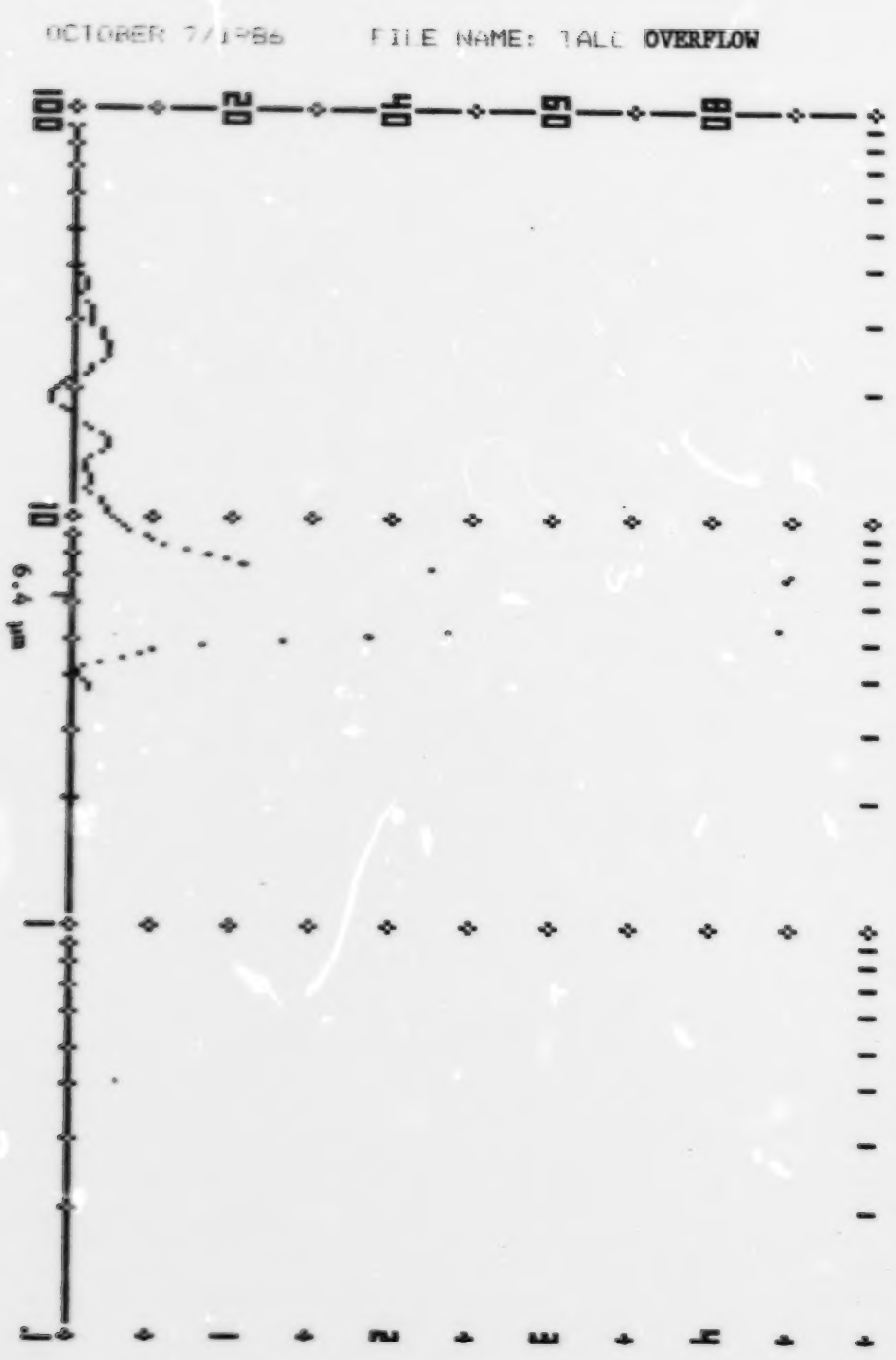
LIQUID: WATER
TEMPERATURE: 26 DEGREES CELSIUS
LIQUID DENSITY: .9968 G/CC
VISCOSITY: .8737 CENTIPOISE

PRINCIPAL PARTICLE DIAMETERS (MICRONS)
STARTING: 50 ENDING: 1.11 MAXIMUM: 42

RUNNING TIME: 1 HR 29 MIN RATE: 466

PARTICLE SIZE (MICRONS)	PERCENT FINER (%)	CHANGE (%)	RATE OF CHANGE (% / INCR)
50	99	1	0
40	99	0	0
30	99	0	.1
20	97	2	-.15
10	96	1	.2
9	96	0	.35
8	93	3	.85
7	89	4	6
6	31	58	6
5	2	29	1.3
4	1	1	0
3	0	1	1.05
2	105	-105	0

END OF DATA


OCTOBER 7/1986
FILE NAME: TALL OVERFLOW
100
90
80
50
10
1
6.4 ms

NOVEMBER 17/1986

OPERATOR: L.MORIN

SAMPLE: TALC UNDERFLOW
SAMPLE DENSITY: 2.92 G/CC

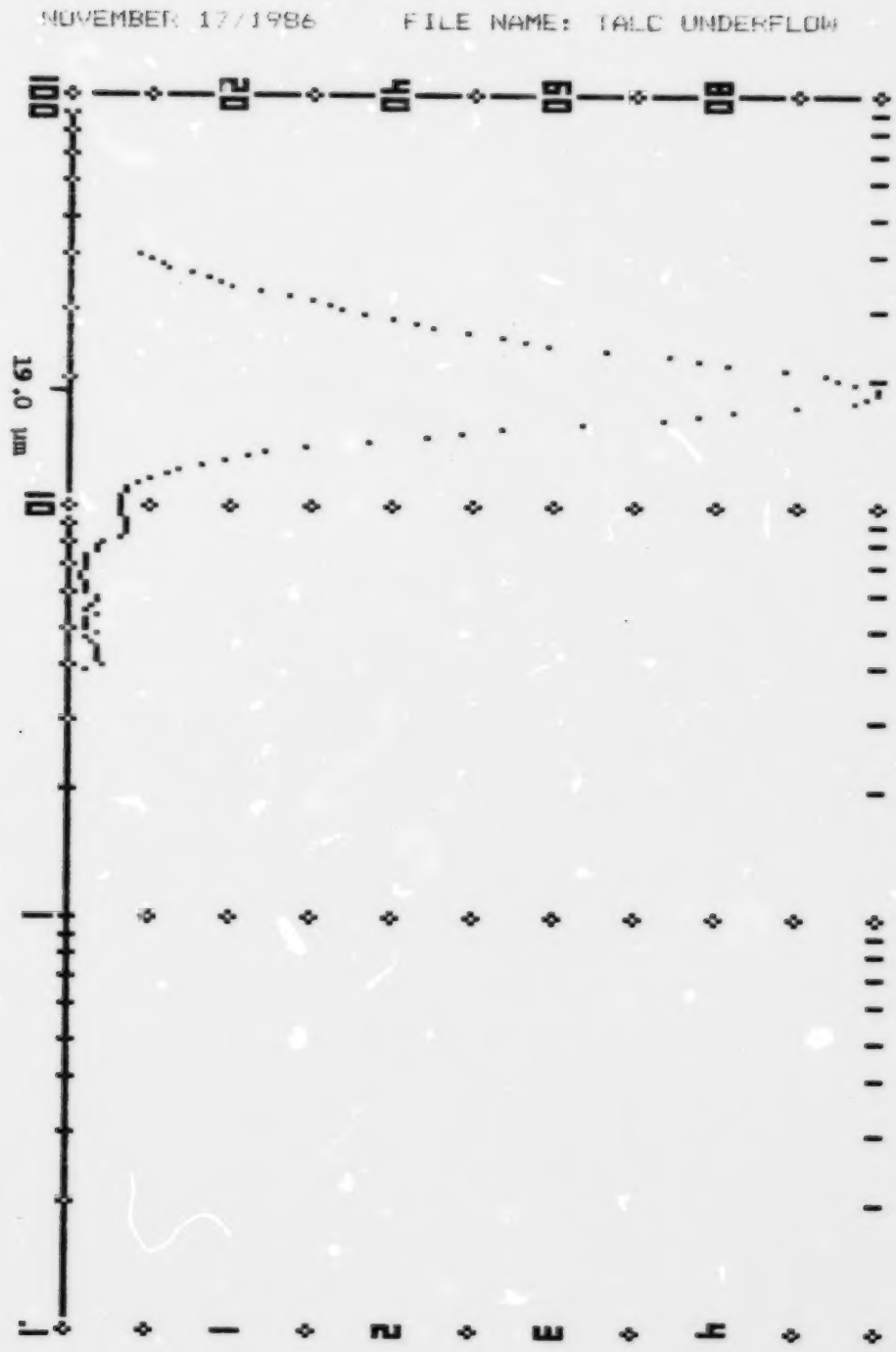
LIQUID: WATER
TEMPERATURE: 28 DEGREES CELSIUS
LIQUID DENSITY: .9963 G/CC
VISCOSITY: .836 CENTIPOISE

PRINCIPAL PARTICLE DIAMETERS (MICRONS)
STARTING: 50 ENDING: .18 MAXIMUM: 41

RUNNING TIME: 1 HR 25 MIN RATE: 487

PARTICLE SIZE (MICRONS)	PERCENT FINER (%)	CHANGE (%)	RATE OF CHANGE (% / INCR)
50	99	1	0
40	98	1	.4
30	90	8	1.65
20	52	38	4.85
10	6	46	.3
9	5	1	.35
8	3	2	.2
7	3	0	.1
6	3	0	.1
5	2	1	.1
4	1	1	.2
3	105	-104	-.5
2	105	0	0
1	105	0	0
.9	105	0	0
.8	105	0	0
.7	105	0	0
.6	105	0	0
.5	105	0	0
.4	105	0	0
.3	105	0	0
.2	105	0	0

END OF DATA


NOVEMBER 17/1986
FILE NAME: TALC UNDERFLOW
100
90
80
60
40
20
10
1
19.0 mm
1
2
3
4

OCTOBER 10/198e

OPERATOR: L.MORIN

SAMPLE: KAOLIN OVERFLOW
SAMPLE DENSITY: 2.6 G/CC

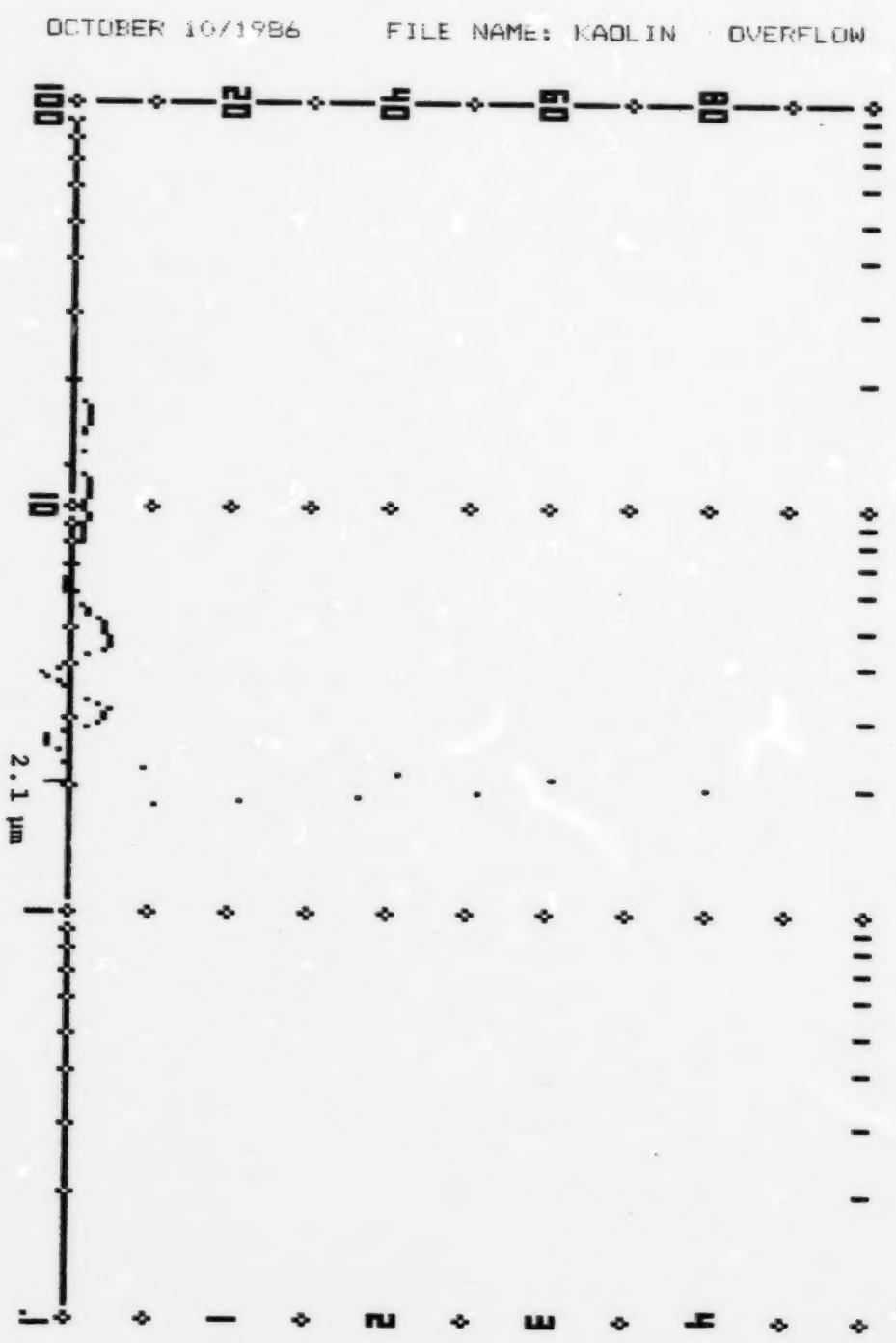
LIQUID: WATER
TEMPERATURE: 33 DEGREES CELSIUS
LIQUID DENSITY: .9947 G/CC
VISCOSITY: .7523 CENTIPOISE

PRINCIPAL PARTICLE DIAMETERS (MICRONS)
STARTING: 28 ENDING: .96 MAXIMUM: 40

RUNNING TIME: 4 HR 50 MIN RATE: 142

PARTICLE SIZE (MICRONS)	PERCENT FINER (%)	CHANGE (%)	RATE OF CHANGE (% / INCR)
20	99	1	0
10	98	1	.05
9	98	0	.1
8	98	0	.05
7	98	0	0
6	98	0	-.05
5	98	0	.2
4	97	1	0
3	96	1	.2
2	96	0	4
1	105	-9	0

END OF DATA


OCTOBER 10/1986 FILE NAME: KAOLIN OVERFLOW
100
02
04
06
08
10
2.1 µm
1
2
3
4

OCTOBER 10/1986

OPERATOR: L.MORIN

SAMPLE: KAOLIN **UNDERFLOW**
SAMPLE DENSITY: 2.6 G/CC

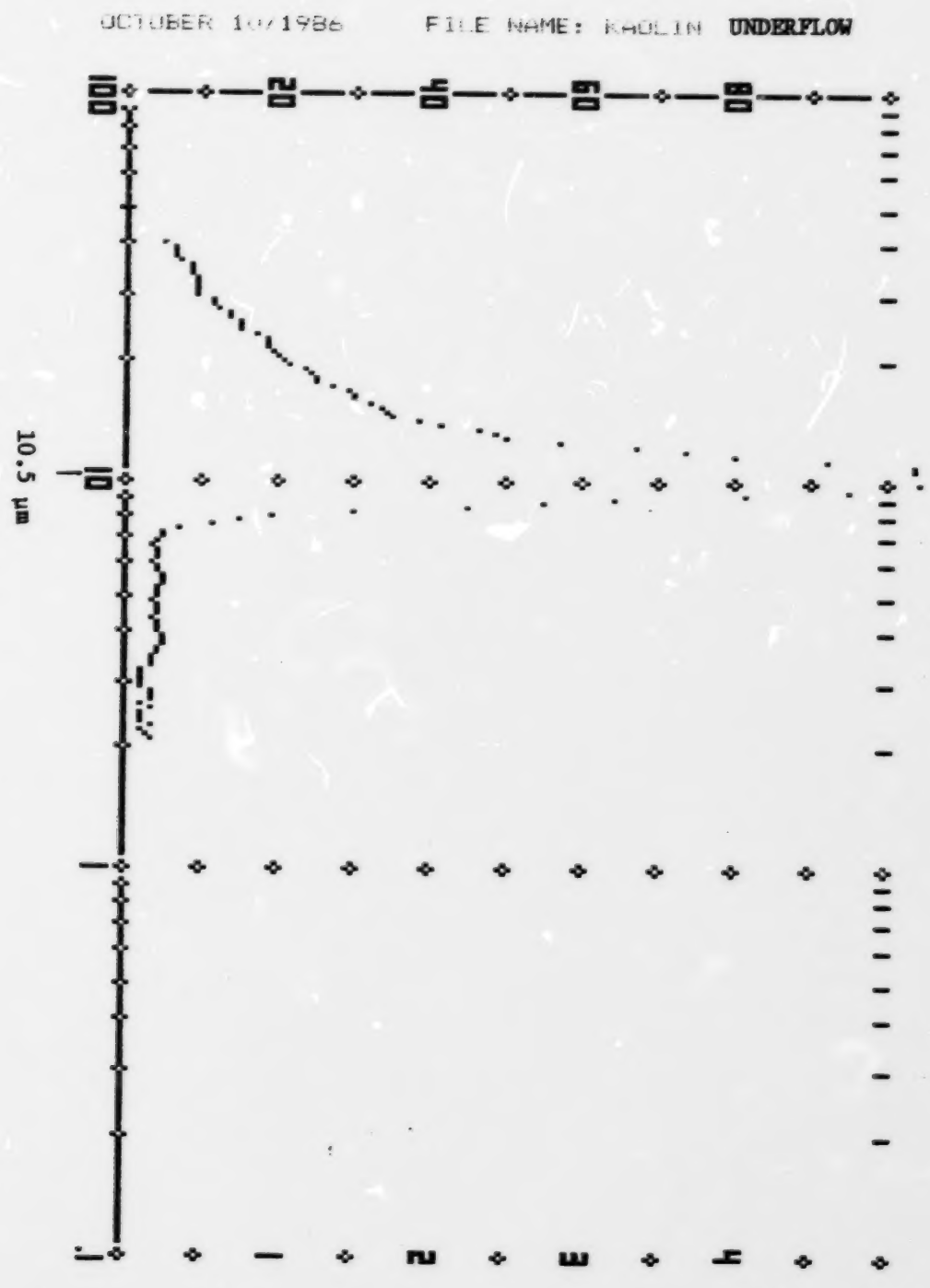
LIQUID: WATER
TEMPERATURE: 28 DEGREES CELSIUS
LIQUID DENSITY: .9963 G/CC
VISCOSITY: .836 CENTIPOISE

PRINCIPAL PARTICLE DIAMETERS (MICRONS)
STARTING: 50 ENDING: 1.18 MAXIMUM: 43

RUNNING TIME: 1 HR 42 MIN RATE: 406

PARTICLE SIZE (MICRONS)	PERCENT FINER (%)	CHANGE (%)	RATE OF CHANGE (% / INCR)
50	97	3	0
40	95	2	.25
30	92	3	.45
20	83	9	1.05
10	33	50	5.45
9	13	20	4.05
8	7	6	.95
7	6	1	.2
6	6	0	.15
5	4	2	.2
4	3	1	.2
3	2	1	.1
2	0	2	-.5

END OF DATA


OCTOBER 10/1986
FILE NAME: KAOLIN UNDERFLOW
100
90
80
50
10
1
10.5 µm